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(54) **A method of making silicone in water emulsions**

Verfahren zur Herstellung von Silicon-in-Wasser-Emulsionen

Procédé de préparation d'émulsions aqueuses de silicone

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(56) References cited:
EP-A- 0 169 098 **EP-A- 0 314 955**
EP-A- 0 417 559 **US-A- 4 248 751**
US-A- 5 035 832

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Description

[0001] The present invention relates to a method of making silicone in water emulsions. Specifically, the present invention relates to a simple process of making silicone in water emulsions in which silicones polymerize by chain extension at the interior of a silicone droplet suspended in water.

[0002] Several techniques are known in the art for the production of silicone in water emulsions (hereinafter 's/w emulsions'). One technique, emulsion polymerization, is described, for example, in European Patent Applications 268,982, 459,500 and 698,633. This process involves emulsifying a low molecular weight silicone and an anionic or cationic surfactant in a high shear inducing device. The silicone is polymerized by the addition of a strong acid or base, often at elevated temperatures. This process can yield relatively high molecular weight silicone polymer (e.g., $<900,000 \text{ mm}^2/\text{sec}$) in water emulsions.

[0003] Emulsion polymerization, however, has a number of drawbacks. For instance, polymerization in the emulsion polymerization process occurs at the silicone water interface. As such, the rate of polymerization is faster with smaller particles because of the larger surface area and, thus, it is impossible to produce large particle size, high molecular weight silicone gum in water emulsions. Similarly, emulsion polymerization involves a number of processing steps and/or materials which are disadvantageous. For instance, emulsion polymerization requires long batch times and caustic materials (strong acidic or basic catalysts which must be neutralized). Finally, the emulsions resulting from emulsion polymerization may have limited utilities because of the materials used in their manufacture. For instance, the anionic and cationic surfactants used in these emulsions can be irritating to the skin and they can affect the stability of products into which the emulsions are incorporated.

[0004] A second technique for the production of s/w emulsions is mechanical emulsion. In this process, a silicone polymer is mechanically emulsified with a variety of surfactants and water. This process allows for the production of anionic, cationic, non-ionic or amphoteric emulsions having a variety of particle sizes and high silicone fractions. Similarly, the process is more advantageous than emulsion polymerization in that the processing time is relatively short and the process does not require heating or a neutralization step.

[0005] Mechanical emulsion, however, is limited by the shear stress which can be induced by the mixer. For instance, conventional mixers are often limited to silicone polymers with a viscosity of about $600,000 \text{ mm}^2/\text{sec}$ at 25°C .

[0006] We have now discovered a more desirable method of making s/w emulsions and novel s/w emulsions having a wide range of physical characteristics.

[0007] The present invention provides in one of its aspects a method of making a silicone in water emulsion, characterised by mixing at least one polysiloxane having reactive end groups, at least one organosilicon material that reacts with said polysiloxane by a chain extension reaction and a metal containing catalyst for said chain extension reaction to form a composition (I), and then mixing composition (I) with at least one surfactant and water, and emulsifying the mixture.

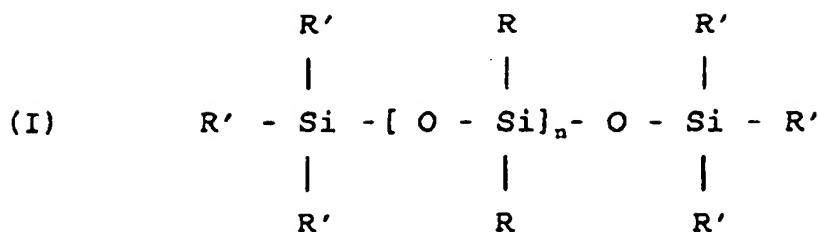
[0008] According to another aspect of the invention, a method of making a silicone in water emulsion, characterised by mixing at least one polysiloxane having reactive end groups, at least one organosilicon material that reacts with said polysiloxane by a chain extension reaction, a metal containing catalyst for said chain extension reaction and a surfactant to form a composition (I'), and then mixing composition (I') with water and emulsifying the mixture.

[0009] The s/w emulsions produced by the process of this invention can have a wide variety of silicone volume fractions, particle sizes and molecular weights including novel materials having large volume fractions of the silicone and large particles containing high molecular weight silicone gums. Moreover, the process results in emulsions in which the particle size and the molecular weight of the silicone inside the droplets are independent parameters.

[0010] The composition (I) containing at least one polysiloxane having reactive end groups, at least one organosilicon material that reacts with said polysiloxane by a chain extension reaction and a metal containing catalyst for said chain extension reaction according to this invention is not critical and nearly any which cures by a chain extension reaction can be used herein. Such chain extension reactions generally involve (1) a polysiloxane which has an end group which reacts with the end group of another polysiloxane or (2) a polysiloxane having a reactive end group which is chain extended with a chain extension agent such as an silane. A small amount of the chain extension can occur at non-terminal sites on the polysiloxane.

[0011] Chain extension reactions with polysiloxanes (polysiloxanes are also called silicones or organopolysiloxanes) are known in the art and can involve, for instance, the hydrosilylation reaction in which an Si-H reacts with an aliphatically unsaturated group in the presence of a platinum or rhodium containing catalyst. Alternatively, the reaction can involve the reaction of an Si-OH (e.g., polymers) with an alkoxy group (e.g., alkoxy silanes, silicates or alkoxy siloxanes) in the presence of a metal containing catalyst. Still other reactions can involve the reaction of an Si-OH with a $\text{CH}_3\text{COOSi-}$ in the presence of water, the reaction of an SiOH with $\text{R}_2\text{C=NOSi-}$, or the reaction of SiOH with an SiH in the presence of a metal containing catalyst.

[0012] The polysiloxane(s) used in the above reactions generally comprises a substantially linear polymer of the structure:



In this structure, each R and R' independently represent a hydrocarbon group having up to 20 carbon atoms such as an alkyl (e.g., methyl, ethyl, propyl or butyl), an aryl (e.g., phenyl), or the group required for the chain extension reaction described above ('reactive group', e.g., hydrogens, aliphatically unsaturated groups such as vinyl, allyl or hexenyl, hydroxys, alkoxys such as methoxy, ethoxy or propoxy, alkoxy-alkoxy, acetoxys, aminos and the like), provided that on average there is between one and two reactive groups (inclusive) per polymer, and n is a positive integer greater than one. Preferably, a majority, more preferably >90%, and most preferably >98% of the reactive groups are end-groups, i.e., R'.

[0013] Preferably n is an integer that results in polysiloxanes with viscosities between about 1 and about 1×10^6 mm²/sec at 25°C.

[0014] If desired, the polysiloxane (I) can have a small amount of branching (e.g., less than 2 mole % of the siloxane units) without affecting the invention, i.e., the polymers are 'substantially linear'. Moreover, if desired, the R and R' groups can be substituted with, for instance, nitrogen containing groups (e.g., amino groups), epoxy groups, sulphur containing groups, silicon containing groups, oxygen containing groups and the like. Preferably, however, at least 80% of the R groups are alkyls and, more preferably, the alkyl groups are methyl groups.

[0015] The organosilicon material that reacts with the polysiloxane by a chain extension reaction can be either a second polysiloxane or a material that acts as a chain extension agent. If the organosilicon material is polysiloxane, it too will generally have the structure described above (I). In such a situation, however, one polysiloxane in the reaction will comprise one reactive group and the second polysiloxane will comprise a second reactive group which reacts with the first.

[0016] If the organosilicon material comprises a chain extension agent, it can be a material such as a silane, a siloxane (e.g., disiloxane or trisiloxane) or a silazane. For instance, a composition comprising a polysiloxane according to the above structure (I) which has at least one Si-OH group can be chain extended by using an alkoxysilane (e.g., a dialkoxysilane or trialkoxysilane) in the presence of a tin or titanium containing catalyst.

[0017] The metal containing catalysts used in the above chain extension reactions are often specific to the particular reaction. Such catalysts, however, are known in the art. Generally, they are materials containing metals such as platinum, rhodium, tin, titanium, copper, lead, etc.

[0018] In a preferred embodiment of the invention, the polysiloxane has at least one aliphatically unsaturated group, preferably an end group, and the organosilicon material is a siloxane or a polysiloxane having at least one Si-H group, preferably an end group, in the presence of a hydrosilylation catalyst. The polysiloxane having at least one aliphatically unsaturated group has the structure (I) wherein R, R' and n are as defined above and provided that on average between one and two (inclusive) R or R' groups comprise an aliphatically unsaturated group per polymer. Representative aliphatically unsaturated groups include vinyl, allyl, hexenyl and cyclohexenyl or a group R"CH=CHR'", where R" represents a divalent aliphatic chain linked to the silicon atom and R'" represents a hydrogen atom or an alkyl group. The organosilicon material having at least one Si-H group preferably has the above structure (I) wherein R, R' and n are as defined above and provided that on average between one and two (inclusive) R or R' groups comprise hydrogen atoms and n is 0 or a positive integer. This material can be a polymer or a lower molecular weight material such as a siloxane (e.g., a disiloxane or a trisiloxane).

[0019] The polysiloxane having at least one aliphatically unsaturated group and the organosilicon material having at least one Si-H group react in the presence of a hydrosilylation catalyst. Such catalysts are known in the art and can include, for example, platinum and rhodium containing materials. These catalysts may take any of the known forms such as platinum or rhodium deposited on carriers such as silica gel or powdered charcoal, or other appropriate compounds such as platonic chloride, salts of platinum and chloroplatinic acids. A preferred material is chloroplatinic acid either as the commonly obtainable hexahydrate or the anhydrous form because of its easy dispersibility in organosilicon systems and its non-effect on colour of the mixture. Platinum or rhodium complexes may also be used e.g. those prepared from chloroplatinic acid hexahydrate and divinyltetramethyldisiloxane. Generally, these catalysts are used in amounts of between about 0.0001 and 10 wt. % based on the weight of the composition (I).

[0020] In a second preferred embodiment of the invention, the polysiloxane has at least one Si-OH group, preferably

an end group, and the organosilicon material has at least one alkoxy group, preferably a siloxane having at least one Si-OR group, or an alkoxysilane having at least two alkoxy groups in the presence of a metal containing catalyst. In this case, the polysiloxane having at least one SiOH group has the structure (I) wherein R, R' and n are as defined above and on average between one and two (inclusive) R or R' groups comprise a hydroxyl group (OH). The organo-
 5 silicon material having at least one alkoxy group can have the structure (I) wherein R, R' and n are as defined above and on average between one and two (inclusive) R or R' groups comprise alkoxy groups, e.g., of the structure (OR) in which R is as defined above and n is 0 or a positive integer. Alternatively, the organosilicon material can be a silane of the structure $R_mSi(OR)_{4-m}$, wherein R is as defined above and m is 0 to 2. Other materials containing the alkoxy group (e.g., alkoxy-alkoxys) may also be used herein.

10 [0021] A variety of metal catalysts for the reaction of an Si-OH with an Si-OR are known in the art and may be employed including, for example, organic metal compounds such as organotin salts, titanates, or titanium chelates or complexes. Examples of catalysts include stannous octoate, dibutyltin dilaurate, dibutyltin diacetate, dimethyltin dineodecanoate, dibutyltin dimethoxide, isobutyl tin tricerate, dimethyltin dibutyrate, dimethyltin dineodecanoate, triethyltin tartrate, tin oleate, tin naphthenate, tin butyrate, tin acetate, tin benzoate, tin sebacate, tin succinate, tetrabutyl titanate,
 15 tetraisopropyl titanate, tetraphenyl titanate, tetraoctadecyl titanate, titanium naphthanate, ethyltriethanolamine titanate, titanium diiso-propyl diethyl acetoacetate, titanium diisopropoxy diacetyl acetate, and titanium tetra alkoxides where the alkoxide is butoxy or propoxy. Generally, these catalysts are used in amounts of between about 0.001 and 10 wt. % based on the weight of the composition (I).

20 [0022] Although a limited number of compounds are exemplified, any composition containing at least one polysiloxane, at least one organosilicon material that reacts with said polysiloxane by a chain extension reaction and a metal containing catalyst for said chain extension reaction can be used herein.

[0023] The mixture used to form the emulsion also contains at least one surfactant (II). This can be a non-ionic surfactant, a cationic surfactant, an anionic surfactant, alkylpolysaccharides, amphoteric surfactants and the like.

25 [0024] Examples of non-ionic surfactants include polyoxyalkylene alkyl ethers, polyoxyalkylene sorbitan alkyl esters, polyoxyalkylene alkyl esters, and polyoxyalkylene alkylphenol ethers, polyethylene glycols, polypropylene glycols, and diethylene glycols.

30 [0025] Examples of cationic surfactants include quaternary ammonium hydroxides such as tetramethylammonium hydroxide, octyltrimethylammonium hydroxide, dodecyl-trimethyl ammonium hydroxide, hexadecyltrimethyl ammonium hydroxide, octyldimethylbenzylammonium hydroxide, decyldimethylbenzyl ammonium hydroxide, didodecyldimethyl ammonium hydroxide, dioctadecyl dimethylammonium hydroxide, tallow trimethylammonium hydroxide and cocotrimethylammonium hydroxide as well as corresponding salts of these materials, fatty acid amines and amides and their derivatives and the salts of the fatty acid amines and amides including aliphatic fatty amines and their derivatives, homologs of aromatic amines having fatty chains, fatty amides derived from aliphatic diamines, fatty amides derived from disubstituted amines, derivatives of ethylene diamine, amide derivatives of amino alcohols, amine salts of long
 35 chain fatty acids, quaternary ammonium bases derived from fatty amides of disubstituted diamines quaternary ammonium bases of benzimidazolines, basic compounds of pyridinium and its derivatives, sulfonium compounds, quaternary ammonium compounds of betaine, urethanes of ethylene diamine, polyethylene diamines and polypropanolpolyethanol amines.

40 [0026] Examples of suitable anionic surfactants include alkyl sulfates such as lauryl sulfate, polymers such as acrylates/C10-30 alkyl acrylate crosspolymer alkylbenzenesulfonic acids and salts such as hexylbenzenesulfonic acid, octylbenzenesulfonic acid, decylbenzenesulfonic acid, dodecylbenzenesulfonic acid, cetylbenzenesulfonic acid and myristylbenzenesulfonic acid; the sulfate esters of monoalkyl polyoxyethylene ethers; alkylphenylsulfonic acid; alkali metal sulfonates, sulfonated glyceryl esters of fatty acids such as sulfonated monoglycerides of coconut oil acids, salts of sulfonated monovalent alcohol esters, amides of amino sulfonic acids, sulfonated products of fatty acid nitriles,
 45 sulfonated aromatic hydrocarbons, condensation products of naphthalene sulfonic acids with formaldehyde, sodium octahydroanthracene sulfonate, alkali metal alkyl sulfates, ester sulfates, and alkarylsulfonates.

50 [0027] Examples of suitable alkylpolysaccharides include, for example, materials of the structure $R^1-O-(R^2O)_m-(G)_n$ wherein R^1 represents a linear or branched alkyl group, a linear or branched alkenyl group or an alkylphenyl group, R^2 represent an alkylene group, G represents a reduced sugar, m denotes 0 or a positive integer and n represent a positive integer as described, for example, in US Patent 5,035,832.

[0028] Examples of suitable amphoteric surfactants include cocamidopropyl betaine and cocamidopropyl hydroxysulfate.

[0029] The above surfactants may be used individually or in combination.

55 [0030] The particle size of the silicone in the emulsion is dependent on, among other factors, the amount and type of surfactant employed. The amount of surfactant used will vary depending on the surfactant, but generally it is used in an amount of between about 1 and 30 wt. % based on the total weight of the composition (I).

[0031] The final material used to form the emulsions herein is the water which forms the continuous phase of the emulsion and into which the silicone oil droplets are dispersed.

[0032] If desired, other materials can be added to either phase of the emulsions. For example, materials which assist in the chain extension reaction (e.g., other chain extenders), conventional inhibitors, perfumes, colorants, thickeners, preservatives, plasticizers, active ingredients (e.g., pharmaceuticals) and the like may be used herein.

[0033] According to the invention, composition (I), surfactant and water are mixed by simple agitation to form a coarse water in oil mixture. This mixture is then emulsified. During emulsification, the coarse water in oil mixture is inverted into a fine silicone in water emulsion. The emulsification can be accomplished by conventional means such as a batch mixer, colloid mill or line mixer. The emulsification process is, thus, simple and fast.

[0034] The composition (I), surfactant and water can be mixed all at once or, alternatively, the materials can be mixed in any order. However, when the polysiloxane, the organosilicon material and the metal containing catalyst of composition (I) are combined, the polymerisation reaction begins. As such, it may be preferred to mix one of the components of the composition (I) last. For example, it may be preferred to mix either the metal containing catalyst or either the polysiloxane or the organosilicon material to the mixture after the other components of composition (I).

[0035] We have discovered that anionic surfactants often increase the kinetics of the chain extension reaction between a polysiloxane having at least one aliphatically unsaturated group and an organosilicon material having at least one Si-H group in the presence of a hydrosilylation catalyst. As such, in a preferred embodiment of the invention wherein these reactants are used, it is often preferred to first mix the polysiloxane containing the unsaturated group and the metal containing catalyst (e.g., the platinum) and then mix in a mixture of the anionic surfactant and the organosilicon material (e.g., the Si-H material). Alternatively, a cure inhibitor could be added to control the reaction kinetics.

[0036] After the above materials are mixed, the water is then added and the silicone phase inverted to form silicone droplets in the water as described above. After inversion, the chain extension reaction continues within the silicone droplet until all the materials have reacted or the reaction has been inhibited.

[0037] The quantity of water and/or surfactant used in the initial phase inversion process may have an impact on the particle size of the final emulsion. For instance, if an emulsion is formed with the same quantity of water in two instances but in the first a large quantity of water is mixed before the phase inversion step and in the second a small quantity of water is mixed before the phase inversion step followed by mixing the remaining additional water after the phase inversion step, the first emulsion will generally have a larger particle size than the second.

[0038] No matter how the water is added (i.e., before and after inversion), the total amount of water used is generally between about 1 and 99 wt. %, preferably between about 6 and about 99 wt. %, based on the weight of the emulsion.

[0039] The polymerisation of the present invention takes place at the interior of the oil droplets by chain extension (i.e., not at the o/w interface) as shown by the fact that the silicone in the droplets generally have the same viscosity as if the silicone is mixed in bulk (i.e., non-emulsified). As such, the degree of polymerisation is not controlled by droplet size, but by the ratio of materials used in the chain extension. This, in turn, allows for the production of a broad range of monodisperse droplet sizes containing polysiloxanes with a high viscosity. Moreover, this technique allow for the production of emulsions with high silicone volume fractions.

[0040] Another of the advantages of the process of the invention is that it can be performed without heat and acidic or basic catalysts in a relatively short time using a wide range of surfactants.

[0041] The emulsions of the present invention can generally have a silicone loading in the range of about 1 to about 94 wt. %. The molecular weight of the silicone can be in the range of about 1 mm²/sec at 25°C to in excess of 10⁸ mm²/sec at 25°C. The mean particle size of the emulsion can vary from about 0.3 to in excess of 100 micrometers. Particularly relevant is the fact that this process produces previously unknown emulsions in which the mean particle size is in the range of about 0.3 micrometers and the viscosity of the silicone is greater than 10⁵ mm²/sec. Specifically, we have discovered emulsions in which the mean particle size is in the range of about 0.3, preferably 1, to 100 micrometers with viscosities of the silicone in the range of 10⁶ to 10⁸ mm²/sec.

[0042] The emulsions of the invention also have a number of other practical advantages. For instance, the emulsions of the invention render the high molecular weight silicone in the droplets easily handleable. Similarly, since the silicone in the droplets of the invention generally have the same viscosity as if the silicone is mixed in bulk, one can determine the viscosity of the silicone before emulsion. Also, this allows one to easily perform quality checks on the silicone in the emulsions.

[0043] The emulsions of the invention are useful in the standard applications for silicone emulsions. Thus, they are useful for personal care applications such as on hair, skin, mucous, teeth, etc. In these applications, the silicone is lubricious and will improve the properties of skin creams, skin care lotions, moisturisers, facial treatments such as acne or wrinkle removers, personal and facial cleansers, bath oils, perfumes, fragrances, colognes, sachets, sunscreens, pre-shave and after shave lotions, shaving soaps and shaving lathers. It can likewise be use in hair shampoos, hair conditioners, hair sprays, mousses, permanents, depilatories, and cuticle coats to provide conditioning benefits. In cosmetics, it function as a levelling and spreading agent for pigment in make-ups, colour cosmetics, foundations, blushes, lipsticks, eye liners, mascaras, oil removers, colour cosmetic removers and powders. It is likewise useful as a delivery system for oil and water soluble substances such as vitamins, organic sunscreens, ceramides, pharmaceuticals and the like. When compounded into sticks, gels, lotions aerosols and roll-ons, the emulsions of this invention

impart a dry silky-smooth payout.

[0044] When used in personal care products, they are generally incorporated in amounts of about 0.01 to about 50 weight percent, preferably 0.1 to 25 wt. percent, of the personal care product. They are added to conventional ingredients for the personal care product chosen. Thus, they can be mixed with deposition polymers, surfactants, detergents, antibacterials, anti-dandruffs, foam boosters, proteins, moisturising agents, suspending agents, opacifiers, perfumes, colouring agents, plant extracts, polymers, and other conventional care ingredients.

[0045] Beyond personal care, the emulsion of the invention are useful for numerous other applications such as textile fibre treatment, leather lubrication, fabric softening, release agents, water based coatings, oil drag reduction, lubrication, facilitation of cutting cellulose materials, and in many other areas where silicones are conventionally used.

[0046] The following Examples are provided so that one skilled in the art will more readily understand the invention. Unless otherwise indicated, all parts and percents are by weight and all viscosities are at 25°C.

Examples 1-3

[0047]

1) 29 parts of a dimethylvinylsiloxyl terminated polydimethylsiloxane having a viscosity of about 7,000 to 12,000 mm²/sec at 25°C; 0.9 parts of a liquid organohydrogenpolysiloxane having the average formula Me₂HSiO (Me₂SiO)₂₀SiMe₂H wherein Me denotes the methyl radical and containing 0.16 to 0.20 % SiH; and 0.015 parts platinum in a platinum catalyst material were mixed.

2) 29 parts of the same dimethylvinylsiloxyl terminated polydimethylsiloxane as above; 1 part of the same organohydrogenpolysiloxane as above; and 0.015 parts platinum as above were mixed.

3) 29 parts of the same dimethylvinylsiloxyl terminated polydimethylsiloxane as above; 1.15 parts of the same organohydrogenpolysiloxane as above; and 0.015 parts platinum as above were mixed.

[0048] To each of the above mixtures was added 0.75 parts Laureth-3 and 1 part Laureth-23. Finally, the following quantities of water were added to the mixtures in 5 stages with mixing after each addition: 1.5 parts, 2.5 parts, 5 parts, 6 parts and 4 parts.

[0049] The resultant emulsions had mean particle sizes of 0.45 micrometers in each case and the viscosity of the silicones in the droplets was as follows:

Ex 1 = 1.25 million mm²/sec

Ex 2 = 5.2 million mm²/sec

Ex 3 = 75 million mm²/sec

Example 4

[0050] Examples 1-3 were repeated except the water was added in 2 stages in the following amounts: 7 parts and 5 parts. The resultant materials had mean particle sizes of 8.5 microns in each case.

Example 5

[0051] Examples 1-3 were repeated except that 12 parts water was added in 1 single stage. The resultant materials had mean particle sizes of 60 microns in each case.

Example 6

[0052] This example demonstrates the use of different surfactants.

[0053] 29 parts of a dimethylvinylsiloxyl terminated polydimethylsiloxane having a viscosity of about 7,000 to 12,000 mm²/sec at 25°C; 1.05 parts of the same organohydrogenpolysiloxane as used in Example 1; and 0.015 parts platinum as in Example 1 were mixed.

[0054] To portions of this mixture was added the surfactant and water identified below:

A. 2 parts sodium laurylether 3 sulfate (anionic)

2 parts water.

Viscosity - 9×10^6 mm²/sec

Mean particle size - 5 micrometers

B. 4 parts acrylates/C10-30 alkyl acrylate crosspolymer

45.2 parts water before inversion

34.8 parts water after inversion

Viscosity - 14.4×10^6 mm²/sec

Mean particle size - 13 micrometers

C. 3 parts decyl glucoside

2 parts water before inversion

34.8 parts water after inversion

Viscosity - 2.03×10^7 mm²/sec

Mean particle size - 2.2 micrometers

D. 6 parts Hexadecylammonium chloride (cationic)

34.8 parts water

Viscosity - 1.55×10^7 mm²/sec

Mean particle size - 5.15 micrometers

E. 2 parts disodium ricinoleamino monoethylamine sulfosuccinate (amphoteric)

6 parts water before inversion

34.8 parts water after inversion

Viscosity - 1.34×10^7 mm²/sec

Mean particle size - 12.2 micrometers

Claims

1. A method of making a silicone in water emulsion, **characterised by** mixing at least one polysiloxane having reactive end groups, at least one organosilicon material that reacts with said polysiloxane by a chain extension reaction and a metal containing catalyst for said chain extension reaction to form a composition (I), and then mixing composition (I) with at least one surfactant and water, and emulsifying the mixture.
2. A method of making a silicone in water emulsion, **characterised by** mixing at least one polysiloxane having reactive end groups, at least one organosilicon material that reacts with said polysiloxane by a chain extension reaction, a metal containing catalyst for said chain extension reaction and a surfactant to form a composition (I'), and then mixing composition (I') with water and emulsifying the mixture.
3. A method according to Claim 1 or Claim 2 **characterised in that** the reactive end groups of the polysiloxane are aliphatically unsaturated groups, the organosilicon material is a polysiloxane having at least one Si-H group and the catalyst is a platinum or rhodium containing catalyst.
4. A method according to Claim 1 or Claim 2 **characterised in that** the reactive end groups of the polysiloxane are Si-OH groups and the organosilicon material is an alkoxysilane having at least two alkoxy groups.
5. A method according to Claim 1 or Claim 2 **characterised in that** the reactive end groups of the polysiloxane are Si-OH groups and the organosilicon material is a polysiloxane having at least one Si-OR group and a metal containing catalyst.
6. A method according to any of the previous Claims **characterised in that** the surfactant is present in an amount of between about 1 and 30 wt. % based on the combined weight of the polysiloxane, the organosilicon material and the metal-containing catalyst.
7. A method according to any of the previous Claims **characterised in that** the water is present in an amount of between about 6 and 99 wt. % based on the weight of the emulsion.
8. A method according to Claim 3, **characterised by**
mixing a polysiloxane having reactive aliphatically unsaturated end groups and a polysiloxane having Si-H

groups with a surfactant to form a first mixture;
 mixing a platinum or rhodium containing catalyst with the first mixture to form a second mixture; and
 mixing water with the second mixture to form the emulsion.

- 5 **9. A method according to Claim 3, characterised by**
 mixing a polysiloxane having reactive aliphatically unsaturated end groups with a platinum or rhodium containing catalyst to form a first mixture;
 mixing an anionic surfactant with a polysiloxane having Si-H groups to form a second mixture;
 mixing the first mixture with the second mixture to form a third mixture; and
 10 mixing water with the third mixture to form the emulsion.
- 10. A method according to Claim 3 characterised by**
 mixing the polysiloxane having aliphatically unsaturated groups with the polysiloxane having Si-H groups
 and the platinum or rhodium containing catalyst to form a first mixture;
 15 mixing the first mixture with a surfactant to form a second mixture; and
 mixing water with the second mixture to form the emulsion.
- 11. A method according to Claim 3 characterised by**
 mixing the polysiloxane having aliphatically unsaturated groups with the polysiloxane having Si-H groups
 and the platinum or rhodium containing catalyst to form a first mixture; and
 20 mixing the first mixture with a surfactant and water to form the emulsion.
- 12. A silicone in water emulsion in which the mean particle size is greater than 0.3 micrometers and the viscosity of the silicone is greater than 10^5 mm²/sec.**
- 25 **13. A silicone in water emulsion according to Claim 12 in which the mean particle size is in the range of about 0.3 to 100 micrometers and the viscosity of the silicone in the range of 10^6 to 10^8 mm²/sec.**
- 30 **14. A silicone in water emulsion made by a method according to any one of Claims 1 to 11.**

Patentansprüche

- 35 **1. Verfahren zur Herstellung einer Silicon-in-Wasser-Emulsion, dadurch gekennzeichnet, dass** mindestens ein Polysiloxan mit reaktiven Endgruppen, mindestens ein Organosiliciummaterial, das mit diesem Polysiloxan durch eine Kettenverlängerungsreaktion reagiert, und ein metallhaltiger Katalysator für diese Kettenverlängerungsreaktion vermischt werden, um Zusammensetzung (I) zu bilden, und dann Zusammensetzung (I) mit mindestens einer oberflächenaktiv Substanz und Wasser vermischt wird und die Mischung emulgiert wird.
- 40 **2. Verfahren zur Herstellung einer Silicon-in-Wasser-Emulsion, dadurch gekennzeichnet, dass** mindestens ein Polysiloxan mit reaktiven Endgruppen, mindestens ein Organosiliciummaterial, das mit diesem Polysiloxan durch eine Kettenverlängerungsreaktion reagiert, ein metallhaltiger Katalysator für diese Kettenverlängerungsreaktion und eine oberflächenaktive Substanz vermischt werden, um eine Zusammensetzung (I') zu bilden, und dann Zusammensetzung (I') mit Wasser vermischt wird und die Mischung emulgiert wird.
- 45 **3. Verfahren gemäß Anspruch 1 oder 2, dadurch gekennzeichnet, dass** die reaktiven Endgruppen des Polysiloxans aliphatisch ungesättigte Gruppen sind, das Organosiliciummaterial ein Polysiloxan mit mindestens einer Si-H-Gruppe ist und der Katalysator ein platin- oder rhodiumhaltiger Katalysator ist.
- 50 **4. Verfahren gemäß Anspruch 1 oder 2, dadurch gekennzeichnet, dass** die reaktiven Endgruppen des Polysiloxans Si-OH-Gruppen sind und das Organosiliciummaterial ein Alkoxysilan mit mindestens 2 Alkoxygruppen ist.
- 55 **5. Verfahren gemäß Anspruch 1 oder 2, dadurch gekennzeichnet, dass** die reaktiven Endgruppen des Polysiloxans Si-OH-Gruppen sind und das Organosiliciummaterial ein Polysiloxan mit mindestens einer Si-OR-Gruppe und ein metallhaltiger Katalysator.
- 6. Verfahren gemäß einem vorausgehenden Ansprüche, dadurch gekennzeichnet, dass** die oberflächenaktive Substanz in einer Menge zwischen etwa 1 und 30 Gew.-%, bezogen auf das vereinigte Gewicht des Polysiloxans,

des Organosiliciummaterials und des metallhaltigen Katalysators, vorhanden ist.

7. Verfahren gemäß einem vorausgehenden Ansprüche, **dadurch gekennzeichnet, dass** das Wasser in einer Menge zwischen etwa 6 und 99 Gew.-%, bezogen auf das Gewicht der Emulsion, vorhanden ist.
8. Verfahren gemäß Anspruch 3, **gekennzeichnet durch**
Mischen eines Polysiloxans, das reaktive aliphatisch ungesättigte Endgruppen aufweist, und eines Polysiloxans, das Si-H-Gruppen aufweist, mit einer oberflächenaktiven Substanz, um eine erste Mischung zu bilden;
Mischen eines platin- oder rhodiumhaltigen Katalysators mit der ersten Mischung, um eine zweite Mischung zu bilden, und
Mischen von Wasser mit der zweiten Mischung, um die Emulsion zu bilden.
9. Verfahren gemäß Anspruch 3, **gekennzeichnet durch**
Mischen eines Polysiloxans, das reaktive aliphatisch ungesättigte Endgruppen aufweist, mit einem platin- oder rhodiumhaltigen Katalysator, um eine erste Mischung zu bilden;
Mischen einer anionischen oberflächenaktiven Substanz mit einem Polysiloxan, das Si-H-Gruppen aufweist, um eine zweite Mischung zu bilden;
Mischen der ersten Mischung mit der zweiten Mischung, um eine dritte Mischung zu bilden, und
Mischen von Wasser mit der dritten Mischung, um die Emulsion zu bilden.
10. Verfahren gemäß Anspruch 3, **gekennzeichnet durch**
Mischen des Polysiloxans, das aliphatisch ungesättigte Gruppen aufweist, mit dem Polysiloxan, das Si-H-Gruppen aufweist, und den platin- oder rhodiumhaltigen Katalysatoren, um eine erste Mischung zu bilden;
Mischen der ersten Mischung mit einer oberflächenaktiven Substanz, um eine zweite Mischung zu bilden, und
Mischen von Wasser mit der zweiten Mischung, um die Emulsion zu bilden.
11. Verfahren gemäß Anspruch 3, **gekennzeichnet durch**
Mischen der Polysiloxans, das aliphatisch ungesättigte Gruppen aufweist, mit dem Polysiloxan, das Si-H-Gruppen aufweist und dem platin- oder rhodiumhaltigen Katalysator, um eine erste Mischung zu bilden, und
Mischen der ersten Mischung mit einer oberflächenaktiven Substanz und Wasser, um die Emulsion zu bilden.
12. Silicon-in-Wasser-Emulsion, in welcher die mittlere Teilchengröße größer als 0,3 μm ist und die Viskosität des Silicons größer als $10^5 \text{ mm}^2/\text{s}$ ist.
13. Silicon-in-Wasser-Emulsion gemäß Anspruch 12, in welcher die mittlere Teilchengröße im Bereich von etwa 0,3 bis 100 μm liegt und die Viskosität des Silicons im Bereich von 10^6 bis $10^8 \text{ mm}^2/\text{s}$ liegt.
14. Silicon-in-Wasser-Emulsion, hergestellt nach einem Verfahren gemäß einem der Ansprüche 1 bis 11.

Revendications

1. Procédé de fabrication d'une émulsion aqueuse de silicone, **caractérisé en ce qu'on mélange au moins un polysiloxane ayant des groupes terminaux réactifs, au moins une matière organosiliciée qui réagit avec ledit polysiloxane par réaction d'extension de chaîne et un catalyseur contenant un métal et catalysant ladite réaction d'extension de chaîne pour former une composition (I) et en ce qu'on mélange ensuite la composition (I) avec au moins un tensioactif et de l'eau et qu'on émulsifie le mélange.**
2. Procédé de fabrication d'une émulsion aqueuse de silicone, **caractérisé en ce qu'on mélange au moins un polysiloxane ayant des groupes terminaux réactifs, au moins une matière organosiliciée qui réagit avec ledit polysiloxane par réaction d'extension de chaîne, un catalyseur contenant un métal et catalysant ladite réaction d'extension de chaîne et un tensioactif pour former une composition (I') et en ce qu'on mélange ensuite la composition (I') avec de l'eau et qu'on émulsifie le mélange.**
3. Procédé selon la revendication 1 ou la revendication 2 **caractérisé en ce que** les groupes terminaux réactifs du polysiloxane sont des groupes insaturés de façon aliphatique, la matière organosiliciée est un polysiloxane ayant au moins un groupe Si-H et le catalyseur est un catalyseur contenant du platine ou du rhodium.

4. Procédé selon la revendication 1 ou la revendication 2 **caractérisé en ce que** les groupes terminaux réactifs du polysiloxane sont des groupes Si-OH et la matière organosiliciée est un alcoxysilane ayant au moins deux groupes alcoxys.
5. Procédé selon la revendication 1 ou la revendication 2 **caractérisé en ce que** les groupes terminaux réactifs du polysiloxane sont des groupes Si-OH et la matière organosiliciée est un polysiloxane ayant au moins un groupe Si-OR et un catalyseur contenant un métal.
6. Procédé selon l'une quelconque des revendications précédentes **caractérisé en ce que** le tensioactif est présent en quantité comprise entre environ 1 et 30 % en poids sur la base des poids combinés du polysiloxane, de la matière organosiliciée et du catalyseur contenant un métal.
7. Procédé selon l'une quelconque des revendications précédentes **caractérisé en ce que** l'eau est présente en quantité comprise entre environ 6 et 99 % en poids sur la base du poids de l'émulsion.
8. Procédé selon la revendication 3, **caractérisé en ce que**
 - on mélange un polysiloxane ayant des groupes terminaux réactifs insaturés de façon aliphatique et un polysiloxane ayant des groupes Si-H avec un tensioactif pour former un premier mélange ;
 - on mélange un catalyseur contenant du platine ou du rhodium avec le premier mélange pour former un deuxième mélange ; et
 - on mélange de l'eau avec le deuxième mélange pour former l'émulsion.
9. Procédé selon la revendication 3, **caractérisé en ce que**
 - on mélange un polysiloxane ayant des groupes terminaux réactifs insaturés de façon aliphatique avec un catalyseur contenant du platine ou du rhodium pour former un premier mélange ;
 - on mélange un tensioactif anionique avec un polysiloxane ayant des groupes Si-H pour former un deuxième mélange ;
 - on mélange le premier mélange avec le deuxième mélange pour former un troisième mélange ; et
 - on mélange de l'eau avec le troisième mélange pour former l'émulsion.
10. Procédé selon la revendication 3 **caractérisé en ce que**
 - on mélange le polysiloxane ayant des groupes insaturés de façon aliphatique avec le polysiloxane ayant des groupes Si-H et le catalyseur contenant du platine ou du rhodium pour former un premier mélange ;
 - on mélange le premier mélange avec un tensioactif pour former un deuxième mélange ; et
 - on mélange de l'eau avec le deuxième mélange pour former l'émulsion.
11. Procédé selon la revendication 3 **caractérisé en ce que**
 - on mélange le polysiloxane ayant des groupes insaturés de façon aliphatique avec le polysiloxane ayant des groupes Si-H et le catalyseur contenant du platine ou du rhodium pour former un premier mélange ; et
 - on mélange le premier mélange avec un tensioactif et de l'eau pour former l'émulsion.
12. Emulsion aqueuse de silicone dans laquelle la taille moyenne des particules est supérieure à 0,3 micromètre et la viscosité du silicone est supérieure à 10^5 mm²/s.
13. Emulsion aqueuse de silicone selon la revendication 12 dans laquelle la taille moyenne des particules est comprise dans l'intervalle allant d'environ 0,3 à 100 micromètres et la viscosité du silicone est comprise dans l'intervalle allant de 10^6 à 10^8 mm²/s.
14. Emulsion aqueuse de silicone fabriquée au moyen d'un procédé selon l'une quelconque des revendications 1 à 11.